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Examining of the segmented electrode use from the viewpoint of the electrolyte volatilizing in molten carbonate fuel cell

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ABSTRACT

Molten carbonate fuel cells (MCFCs) have entered the pre-commercialization phase, and have been experimentally demonstrated in real world applications, including beer brewery, etc. However, though MCFCs have a high potential and an enough operating experience as an energy supply system, they are not explosively widespread. One of these reasons is cost of cell components. Because the thickness of both electrodes is 0.8 mm and both electrodes are made of porous plates of 1 m² of the electrode area, they are often broken by a thermal stress in the sintering process of an electrode and by a worker's carelessness at the cell assembly process. Generally, because these cracking electrodes can potentially cause electrolyte leakage and gas crossover, they are not used to a MCFC stack and are disposed of. Therefore, it made the cost of MCFC be raised. The performance of a cell that uses a mosaic electrode has been evaluated. However, the causal relation between the cracking of an electrode and an electrolyte-leakage has not been yet confirmed. If this causal relationship is elucidated, a cracking electrode or a mosaic electrode can be used to MCFC, such that the cost of MCFC systems would consequently decrease. Therefore, we studied the causal relation between the cracking of an electrode and electrolyte leakage and gas crossover using a visualization technique. In the case of an anode electrode where the centre section of a cell has crack of about 1 mm, the electrolyte leakage from this crack could not be observed by the visualization technique. Moreover, the gas crossover could not be also observed by the visualization technique, and nitrogen in the anode exhaust gas was not detected by a gas chromatography. However, the electrolyte leakage observed from the wet-seal section though the gap between the separator and the electrode was always 1 mm or less. Therefore, electrolyte leakage hardly occurs, even if a cracked anode electrode is installed into the centre section of the cell. On the other hand, although the volatile substance gushes from the wet seal section, the electrolyte leakage/volatilization phenomenon does not occur at the centre of the cell or at the gap between each segmented cathode. The volatile substance in the cathode gas-distributor-channel is composed of the electrolyte mist and the electrolyte volatile substance, and the rate of release is about 2.5 times that of anode side. Although the segmented electrode can be applied to the anode in a MCFC, it cannot be applied to a cathode from the viewpoint of the electrolyte leakage/volatilization.

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1. Introduction

Molten carbonate fuel cells (MCFCs) have energy efficiencies better than low temperature fuel cells, and are expected to become an alternative source of large-scale power generation, such as that of thermal power plants. A stable operation of 40,000 h is one of the requirements for the commercial use of MCFCs. Although Tanimoto et al. succeeded in operating a small-sized MCFC for 40,000 h, electrolyte was added 16 times for long-term operation, because the cell performance decreases with a decrease in the electrolyte [1]. From this result, it is clear that the electrolyte disappears as both a gas and a liquid during operation. Although the leakage of electrolyte in the liquid phase can be prevented, the leakage in the gas phase has not been investigated, despite it being such a serious problem. We have been studying the volatile electrolyte phenomenon in MCFCs using a non-contact image measurement technique since 2004 [2–4]. The behaviour of vaporized molten carbonates in both the cathode and the anode gas-distributor-channel is elucidated under MCFC operating conditions.

Because MCFCs are composed of thin, large-area (about 1 m^2) electrodes and electrolyte tiles, a large-scale facility that makes the cell components is necessary. Moreover, because both electrodes and the electrolyte tile, which are thin porous plates, are easily broken during the sintering process and the cell assembly process, a poor yield of these cell components raises costs. From the viewpoint of the cost of MCFCs, if the yield of the electrodes can be improved

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or if broken cell components can be used, the cost of MCFCs can be reduced. Although Kuroe et al. have studied electrolyte migration by multi-electrode [5,6], this differs from our idea as their segment electrodes were separated perfectly by a separator that has four windows. Moreover, because a cracking electrode has the potential to cause electrolyte leakage and gas crossover, it has not been used until now. In addition, it has been said that the mosaic electrode will easily cause the shrinkage. However, the shrinkage can be avoided by adding aluminium for shrinkproofing. It is necessary to examine the application of the mosaic electrode to MCFC. Therefore, we studied the causal relation between the cracking of an electrode and electrolyte leakage and gas crossover using a visualization technique. This paper is composed of three experiments, as follows. First, the validity of the mosaic electrode is verified by comparing the standard performance of the cell in which mosaic electrode is incorporated with that of a standard cell. Second, the electrolyte volatilization from the gap between each segmented electrode is evaluated by visualization of a cell in which two-segmented anodes are installed. Third, the electrolyte volatilization from the gap of each segment electrode is evaluated by visualization of a cell in which two segmented cathodes are installed.

2. Evaluation of the standard performance of the cell with mosaic electrode

Fig. 1 shows a photograph of the cell frame in which the mosaic electrode is installed. The mosaic electrode is made by dividing a conventional electrode into nine segments. Here, only one electrode was divided into nine, while the other was a conventional electrode. Moreover, the gap of each segmented electrode is from 0.2 to 0.3 mm. MCFC components are made by the National Institute of Advanced Industrial Science and Technology (AIST). Anode and cathode materials are conventional, and the electrolyte is a $(52 + 48) \text{ mol}\% (\text{Li} + \text{Na})/\text{CO}_3 \text{ melt}$. The electrolyte matrix is a lithium aluminium oxide, and the electrode area is 16 cm². Generally, as the single cell made by AIST with Li/Na electrolyte system achieves stabilized performance after one week of the operation, we define the cell performance after one week of the operation as the initial performance. Fig. 2 shows the initial *I–V* performance of the cell with nine segmented electrodes of anode or cathode at steady state



Fig. 1. Photograph of the cell frame in which mosaic electrode is installed.



Fig. 2. Comparison of initial *I*–*V* performances of the cell with nine-segmented electrodes with that of the conventional cell.

period (about one week after operation). The initial cell voltage of our standard cell under 150 mA cm⁻² of current density is from 780 to 820 mV, the initial internal resistance is from 25 to 33 m Ω , and the initial nitrogen concentration of anode exhaust gas under OCV, as an indicator of gas crossover, is 1% or less, respectively. As shown in Fig. 2, the initial performance of the cell with nine-segmented anode is almost same as that of standard cell. However, the performance of the cell with nine-segmented cathode is worse than that of other because the nitrogen concentration of the cell with the nine-segmented cathode is higher than that of other. The life performance of the cell with the nine-segmented anode is almost the same as that of the conventional cell, and is guite stable, as shown in Fig. 3. However, the life performance of the cell with the nine-segmented cathode suddenly deteriorated when the operation time exceeded one week. The cell resistance and the nitrogen concentration in the anode exhaust gas increase with this performance degradation. To examine the influence of the gas crossover in detail, the nitrogen concentration in the anode exhaust gas was measured when pure oxygen was supplied to the cathode instead of air. If nitrogen in the anode exhaust gas originates the fact that the cathode gas leaks to the anode side with the gas crossover, the nitrogen concentration in the anode exhaust gas has to decrease if pure-oxygen was supplied to the cathode instead of air. In a word, the nitrogen concentration of several percentages has to decrease to 1% or less when pure-oxygen is supplied to the cathode instead of air. However, if the nitrogen concentration in the anode exhaust gas



Fig. 3. Life performances of the cell with nine segmented electrodes.

does not decrease even if pure-oxygen is supplied to the cathode instead of air, nitrogen on the anode side originates in the entrainment of air from not the crossover origin but the wet seal. Therefore, we judge that the gas crossover is not caused if the nitrogen concentration in the anode exhaust gas does not decrease until 1% or less even if pure-oxygen is supplied to the cathode instead of air. As a result, about the cell with nine-segmented cathode, because the initial nitrogen concentration decreases from 0.558% to 0.344% by exchanging the cathode supply gas from air into pure oxygen, the gas crossover is not occurred. However, the cell voltage decreases with the increase of the nitrogen concentration when the operating time exceeds 200 h. Although this nitrogen concentration is reduced by half by exchanging the cathode supply gas from air into pure oxygen, it does not decrease until 1% or less. Moreover, the reduction amount of the nitrogen concentration according to the exchange of cathode gases is almost constant, and these nitrogen concentrations increase. Therefore, not only the gas crossover but also the entrainment of air from the wet seal is caused, the contribution of the entrainment of air from the wet seal is larger than the crossover. Therefore, in the performance degradation, the inclusion of air from the wet-seal section is a potential cause. One reason why air mixed into the cell from the wet-seal section might be that the gastight of the wet-seal section could not be kept as the loading electrolyte decreases by the leakage/volatilization of an electrolyte. In this case, there is a possibility that the electrolyte leaks from the gap between each segmented cathode. Therefore, the electrolyte leakage from the gap of each segmented electrode has to be verified by a visualization experiment.

3. Verification of the electrolyte leakage/volatilization phenomenon by a visualization technique

3.1. Experimental apparatus and procedure for the visualization experiment

Fig. 4 shows the experimental apparatus, and Fig. 5 shows a schematic diagram of the structure of a cell frame with two observation windows and an irradiation window. The irradiation window is installed on the front of the cell on the gas inlet side; the observation window to observe the electrode midrange is installed on the side midrange of the cell, and the observation window to observe the wet-seal section is installed on the side upstream part of the cell. Cell voltage is measured and recorded using a data logger, and the image of the volatile phenomenon is shown by irradiating a YAG laser light from an irradiation window into the channel; measurements are made from the two observation windows with a high spatial resolution video camera (12 bits). Images, 1300×1030 pixels in resolution, were saved directly into computer memory. In the case of a supply gas condition, the anode standard gas is 70.4 $H_2/17.6 CO_2/12 H_2O$ (vol.%), and the cathode standard gas is 70 air/30 CO₂ (vol.%), with each gas' utilization being 40%. To verify the electrolyte leakage from the gap between each segmented electrode, the difference with the above-mentioned mosaic cell is as follows:

- (1) To examine the influence of differences in gap composition, anode/cathode electrodes are divided in half. This means that the observation window of the midrange can be used to observe the electrolyte leakage/volatilization from the gap between each segmented electrode, while the observation window of the wet-seal section can be used to observe it from the gap between the electrode and the metal cell frame.
- (2) The current collector is installed only in the parts outside the optical path of the laser, to prevent from diffuse reflectance from the laser light and to protect the CCD camera.



Observation for centre section



- (3) Because the cell performance of the mosaic cell with the nine-segmented anode was good, the gap size between each segmented anode is about 1 mm, to close up the electrolyte leakage/volatilization from this gap.
- (4) Conversely, because the cell performance of the mosaic cell with nine-segmented cathode is bad, the gap size between each segmented cathode is as small as for the above-mentioned mosaic cell.
- (5) In a conventional cell, because the electrolyte volatilization amount is a little, the volatile phenomenon cannot be observed by the visualization technique and it is difficult to collect the amounts of the volatile substances for analysis. Therefore, in the visualization cell, the electrolyte-loading amount of the



Fig. 5. Schematic diagram of the visualization cell.



Fig. 6. Initial *I–V* performances of the visualization cells.

visualizing electrode is twice of a conventional electrode to promote the volatilizing phenomenon.

3.2. Results and discussion

Fig. 6 shows the I-V performance of the visualization cells, the cell resistance, and the nitrogen concentration of the anode exhaust gas under open-circuit-voltage (OCV) conditions during a steady state period (about one week after operation). The visualization cell performances are 260–320 mV worse than the standard cell performance and the visualization cell performance with a two-segmented anode is about 60 mV worse than that with a two-segmented cathode. The reason why cell resistance of the visualization cells are higher than that of the standard cell is that the

contacted part of the visualization cell frame and the electrode is small, as shown in the above-mentioned difference (2). Moreover, the visualization cell performances are worse than the standard cell performance because the gas diffusion resistance of the visualization cell is larger than that of a conventional cell, as shown in the above-mentioned difference (5). The nitrogen concentration of the anode visualization cell is larger than the standard and the cell with the nine-segmented anode (refer to Fig. 2) are. This is due to the above-mentioned difference (3), and the possibility is high that the gas crossover occurs at the 1 mm of gap. Moreover, because the nitrogen concentration also exceeds 1% even if pure oxygen is supplied to the cathode, air mixes into the cell from the wet-seal section. There is a possibility that the gap between the electrode and the metal cell frame becomes large by shifting or shrinking of the segmented anode during the start-up procedure.

On the other hand, the nitrogen concentration of the cathode visualization cell is larger than that of the standard cell, and is the same as the cell with the nine-segmented cathode. Because the nitrogen concentration decreases until 0.415% when pure oxygen is supplied to the cathode, the inclusion of air from the wet-seal section into the cell did not occur. Therefore, the reason for the performance degradation is the above-mentioned differences (2) and (4). There is a possibility that the gas crossover from the gap section without a current collector (observation area) was the cause. These hypotheses are confirmed by the visualization experiment.

3.2.1. Visualization of anode gas-distributor-channel

The electrolyte leakage/volatilization phenomenon of the gap between each segmented electrode is elucidated by observing the centre section of the cell (i.e., the gap of the two-segmented anode). Fig. 7 (a) and (b) show the measurement image of the centre section in the anode gas-distributor-channel immediately after operation under OCV and using 75 mA cm⁻² of current density. In addition, Fig. 7(c) and (d) shows the centre section at one week after



Fig. 7. Measurement image of the centre section in the anode gas channel.



Fig. 8. Change of the concentration of volatile substance with the current cycle.

operation under OCV and using 100 mA cm^{-2} of current density. Here, the current density is set to the maximum value ((b): 75 mA cm^{-2} , (d): 100 mA cm^{-2}) that can be taken out of MCFC at the time of the experiment to clarify the difference of the influence on the phenomenon of no load and on-load. Moreover, to confirm the electrolyte leakage/volatilization phenomenon, the measurement image is displayed as a negative image and each image is the integration of five images taken over one second. The gap of 1 mm is located under the centre of the measurement image, the gas inlet is located on the left side, and the gas outlet is located on the right side. Although there is some transient volatility in the bulk flow immediately after operation starts under OCV conditions (refer to Fig. 7 (a)), volatile substance or gushed substance from the gap cannot be observed even if the cell is generating electricity (refer to Fig. 7 (b)). After one week, however, volatile substance

cannot be observed in the bulk flow under OCV conditions (refer to Fig. 7 (c)), a large amount of volatile substance appears in the bulk flow when generating electricity (refer to Fig. 7(d)). However, the electrolyte leakage/volatilization from the gap between each segmented anode does not occur, even if the operation time exceeds one week and the MCFC generates electricity. Moreover, because the flow of this volatile substance does not fluctuate even if the volatile substance in the bulk flow flows on the gap, gas crossover and electrolyte leakage are not caused. Here, to confirm the dependency of a large amount of volatile substance evolution to the power generation, the volatile substances in the bulk flow are observed according to the following current cycle; (1) the electric current is intercepted after the current density is kept at 100 mA cm⁻² for 12 s. (2) Afterwards, the state of OCV is maintained for 26 s. (3) The power generation (100 mA cm⁻² of the current density) is restarted. Moreover, the mean intensity is obtained by image processing of the measurement images to clearly identify the dependency of evolution of a large amount of volatile substance on power generation. Here, because the range of the intensity is a gradation from 0 to 255, the concentration of volatile substance is defined by dividing the mean intensity obtained from an image processing by 255. Fig. 8 shows the change of the concentration of volatile substance with the above-mentioned current cycle. It is clear that the release of the volatile substance depends on the cell reaction because the mean intensity decreases with the decrease of the current density. However, the electrolyte leakage/volatilization phenomenon is not caused, or cannot be observed, under OCV conditions. Therefore, we presume that the volatile substances in the bulk flow are produced by reacting with an electrolyte to CO₂ generated by the cell reaction.

On the other hand, Fig. 9 shows the measurement image of the wet seal section in the anode gas-distributor-channel. Here, Fig. 9(a), (b), (c) and (d) shows the measurement image immediately



Fig. 9. Measurement image of the wet seal section in the anode gas channel.



Fig. 10. Measurement image of the centre section in the cathode gas channel.

after operation under OCV conditions, immediately after operation under 125 mA cm⁻² of current density, at one week after operation under OCV conditions and at one week after operation under 125 mA cm⁻² of current density. The volatile substances gush from the wet seal section constructed between anode and cell frame. as shown in Fig. 9 (a), where this gush is promoted, and its particle size increases, with the increase in current density, as shown in Fig. 9(b). This gush originates because the electrolyte of the wet seal section flows as a mist with the supplied gas, or with the H₂O and CO₂ generated in the anode cell reaction. After one week, this gush decreases considerably under OCV conditions (refer to Fig. 9(c)), the amount of this gush decreases and the particle size of this gush becomes small under 125 mA cm⁻² of current density (refer to Fig. 9(d)). Therefore, we presume that the gush from the wetseal section immediately after operation begins is an electrolyte mist, and at one week after operation is a volatilizing electrolyte. To confirm this hypothesis, these volatile substances were collected by a membrane filter in the sampling probe inserted in the cell midrange for six days, and analyzed by ion chromatography. As a result, the composition of the volatile substances was found to be 41 Li₂CO₃/59 Na₂CO₃ (the result of conversion molar concentration of Li/Na ions into molten salt), and the volatile rate is about 4.5×10^{-9} mol day⁻¹ cm⁻² (this is calculated by the analyzed result, the sampling time and the electrode area). Here, because the electrolyte-loading amount in the visualizing anode electrode is twice of a conventional electrode to promote the volatilizing phenomenon, the electrolyte volatile rate of the actual MCFC is less than 4.5×10^{-9} mol day⁻¹ cm⁻². Although this composition is close to that of the electrolyte (52 Li₂CO₃/48 Na₂CO₃), it shifts to the Na side. Therefore, we understand that this volatile substance is composed of the electrolyte and sodium hydroxide. These mist electrolytes volatilize in the gas (we assume that the origin of this mist is sodium hydroxide [2,3]), are changed into a powder of Na₂CO₃ by reacting with CO₂, and become a smaller mist due to the gas flow. The electrolyte leakage/volatilization phenomenon in the anode gasdistributor-channel can be characterized from the current results, as follows:

(1) This volatile substance is an electrolyte gushed from the wetseal section until the electrolyte is loaded into both electrodes



Fig. 11. Measurement image of the wet seal section in the cathode gas channel.

and the electrolyte matrix. This leaked electrolyte flows downstream and is observed at centre of the cell immediately after operation starts. Because this volatile substance is almost identical with electrolyte and hardly reacts with the CO₂ generated by the cell reactions, the volatile substance is barely observed at the centre of the cell.

(2) After one week, because the electrolyte gush from the wetseal section decreases considerably under OCV conditions, the volatile substance is hardly observed at the centre of the cell. However, because the electrolyte volatilizes as a sodium hydroxide from the wet-seal section with the cell reaction and flows downstream, it is observed as a large amount of volatile substance by reacting with the CO₂ generated by cell reaction at centre of the cell.

From the viewpoint of the segmented anode, the gap between the electrode and the cell frame becomes large and the electrolyte leakage from this gap is caused by which each segmented electrode shifts or shrinks if the gap between each segmented electrode was 1 mm or more. However, although the segmented anode is effective in reducing costs if the gap between each segmented anode could be suppressed to 0.3 mm or less (the gap of the nine-segmented electrode is about 0.2–0.3 mm), it is necessary to examine the tolerance of the gap where the electrolyte leakage is not caused.

3.2.2. Visualization of cathode gas-distributor-channel

The segmented cathode is examined in a similar method as for the anode. Fig. 10(a) and (b) shows the measurement image of the centre section in the cathode gas-distributor-channel under OCV and $125 \,\mathrm{mA}\,\mathrm{cm}^{-2}$ current density immediately after operation begins; Fig. 10(c) and (d) shows it after one week of operation, and Fig. 10(e) and (f) shows it after two weeks of operation. In addition, Fig. 11 shows the measurement image of the wet seal sec-

tion in the cathode gas-distributor-channel, and Fig. 11(a) and (b) shows it under OCV and 125 mA cm⁻² of current density immediately after operation begins, Fig. 11(c) and (d) shows it under OCV and 125 mA cm⁻² of current density after one week of operation. From these figures, the electrolyte leakage/volatilization phenomenon of cathode side is a little different from that of anode side. This gush hardly depends on the cell reaction, as shown in Fig. 11(a) and (b). The electrolyte leakage/volatilization from the gap cannot be observed as shown in Fig. 11 because the gap between each segment is very narrow. Moreover, the volatile substance is collected in the membrane filter through the sampling probe inserted in the cell midrange for six days, and is analyzed by ion chromatography. As a result, the composition of the volatile substance is 37 Li₂CO₃/63 Na₂CO₃ (mol%), and the volatile rate is about 11.8×10^{-9} mol day⁻¹ cm⁻². Here, because the electrolyte-loading amount in the visualizing cathode electrode is twice of a conventional electrode to promote the volatilizing phenomenon, the electrolyte volatile rate of the actual MCFC is less than 11.8×10^{-9} mol day⁻¹ cm⁻². The sodium content and the volatile rate are considerably more than that of the anode. As compared to the electrolyte gush from the wet-seal section (refer to Figs. 9 and 11), the gush of the cathode side is more than that of the anode side. In addition, we understand that a sodium element in the effusion electrolyte from each gap becomes volatile selectively with the supplied gas because the amount of cathode supply gas is about twice that of the anode side. Because NaOH is in a gas phase if the sodium element becomes volatile as NaOH, this NaOH cannot be observed by the visualization technique. This is one reason why the sodium content of the analyzed result is larger than the lithium content. Moreover, the reason that the drifting electrolyte in the bulk flow decreases with the increase of current density is that the gas is absorbed into the electrode because the cathode reaction is a consumption system.

From the above-mentioned result, we understand that the electrolyte leakage/volatilization phenomenon in the cathode channel is caused as follows:

- (1) Because the volatile substance gushes from the wet seal section as well as from the anode side and it flows to downstream gradually, as shown in Fig. 11(a) and (b), the gushed electrolyte is not reached to the centre of the cell immediately after operation starts, as shown in Fig. 10(a) and (b).
- (2) Though the electrolyte gush from the wet-seal section ends after one week, as shown in Fig. 11(c) and (d), this volatile substance is not easily exhausted and is accumulated in the cell midrange as shown in Fig. 10(c) and (d).
- (3) Most of the drifting electrolyte at centre of the cell is vented to the outside, as shown in Fig. 10(e) and (f), and the cell reaches a stable state, such that the electrolyte leakage/volatilization phenomenon is hardly caused.

From these results, the electrolyte leakage/volatilization phenomenon of the cathode side can be suppressed if the supply gas and the current density are set low for one week after the start-up procedure.

From the viewpoint of the segmented cathode, dividing the cathode electrode enlarges the gap of the wet-seal section and electrolyte leakage/volatilization is promoted. The segmented cathode might be applicable to MCFCs if the assembly accuracy of the electrode and the separator is good (if the gap between electrode and cell frame is kept 0.5 mm or less). Therefore, it is necessary to observe the above-mentioned notes and to use the segmented cathode, and it is necessary as well as anode side to examine the tolerance of the gap where the electrolyte leakage is not caused. Moreover, the segmented electrode has to be evaluated by long-term operation.

4. Conclusion

The objective of the study is examining of segmented electrode use from viewpoint of the electrolyte volatilizing in MCFCs. The obtained results in this study are summarized as follows:

- (1) Because the cell performance with the segmented anode is almost the same as that of a conventional anode, the segmented anode can be applied to reduce the cost of MCFCs.
- (2) Most of the volatilizing phenomenon of an electrolyte is caused at the wet-seal section immediately after the start-up, and it ends after about one week.
- (3) The composition of the volatile substances in anode channel is 41 Li₂CO₃/59 Na₂CO₃ (mol%), and most of the volatile substances originate in the electrolyte leakage from the wet-seal

section until about one week after operation. After one week, most of the volatile substances originate in the electrolyte volatilization.

- (4) The amount of the electrolyte leakage from the wet-seal section of anode side depends on the current density.
- (5) Although the volatile substance gushes from the wet seal section, the electrolyte leakage/volatilization phenomenon does not occur at centre of the cell and the gap of two-segmented anode/cathode. However, if the number of the segmented part increases and the gap is grown, the electrolyte leakage/volatilization phenomenon might be caused at centre of the cell. It is necessary to examine the tolerance of the gap where the electrolyte leakage is not caused.
- (6) Although the volatile substance in the cathode gas-distributorchannel is composed of the electrolyte mist and the volatilized electrolyte and the rate of release is about 2.5 times that of anode side, these volatile substances are not observed until two weeks after operation.
- (7) Though the segmented cathode might be applicable to MCFCs if the assembly accuracy of the electrode and the separator is good, the application of segmented cathode to MCFCs has to be examined further.
- (8) The electrolyte leakage/volatilization phenomenon of the cathode side can be suppressed if the supply gas and the current density are set low for one week after the end of the start-up procedure.

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